

DONOR-ACCEPTOR CONJUGATED COOLIGOMERS FOR SINGLE MOLECULE SOLAR CELLS*

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Abstract Five novel donor-acceptor (D-A) conjugated cooligomers (**F4B-hP**, **F5B-hP**, **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP**) were synthesized. The absorption spectra of the cooligomers cover a wide range from 300 nm to 630 nm. The cooligomers could form films featured by alternating D-A lamellar nanostructures with the periods relative to the molecular lengths after thermal annealing or solvent vapor annealing. Single molecule solar cells were fabricated, and **F5B-hP** exhibited the best device performance. When the film of **F5B-hP** was thermally annealed, a power conversion efficiency (PCE) of 1.56% was realized. With solvent vapor annealing, the PCE could be further improved to 1.72% with a short-circuit current (J_{SC}) of 5.76 mA/cm², an open-circuit voltage (V_{OC}) of 0.87 V and a fill factor (FF) of 0.34.

Keywords: Single molecule solar cells; Monodisperse conjugated oligomers; Lamellar nanostructures; Bulk heterojunction solar cells.

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INTRODUCTION

Since the exciton diffusion length of organic semiconductors is *ca.* 10 nm, a fine phase separation of electron donor (D) and acceptor (A) is necessary to achieve efficient charge separation in organic solar cells (OSCs)^[1]. Meanwhile, the formation of bicontinuous D and A phases is essential for efficient charge carrier transport. Physical blending of D and A materials has been widely used for fabricating OSCs^[2–6]. However, the ideal morphology is hard to be realized for the blend films of D and A materials even after thermal annealing, solvent vapor annealing or with the addition of additives. D-A block copolymers are known to form well-defined nanostructures easily due to the phase separation of D and A blocks^[7]. Recently, many efforts had been made to synthesize D-A block copolymers by covalently attaching perylenediimide (PDI) derivatives^[8–12] or fullerene derivatives^[13–18] to one block of the copolymers as side substituents. However, due to the polydispersity of the polymers, the morphology was generally of low degree of order with the domain size larger than 20 nm^[19].

Monodisperse conjugated oligomers are ideal building blocks for well-defined nanostructures due to their uniform chemical structures and easily tuned molecular length^[7, 20, 21]. We recently reported that D-A conjugated cooligomers (*e.g.* **F5T8-hP**) comprising oligo(fluorene-*alt*-bithiophene)s (OFbTs) and PDIs as the D and A

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segments, respectively, could form D-A lamellar nanostructures with the periods relative to the molecular lengths after solvent vapor annealing for making efficient single molecule solar cells^[22, 23]. However, the absorption of the cooligomers only covered the range of 300–550 nm, which significantly limited the efficient sunlight harvesting, and resulted in a rather low short-circuit current. To address this issue, in the current paper, an electron-deficient unit 2,1,3-benzothiadiazole (BT), which is widely used for constructing low band gap conjugated polymers^[24–26], was introduced into the OFbT D segment to lower its band gap. Five D-A cooligomers, *i.e.* **F4B-hP**, **F5B-hP**, **F5B2[1,2]-hP**, **F7B2[1,2]-hP** and **F5B2[1,3]-hP**, were synthesized, and their self-assembly and photophysical properties were studied in detail.

EXPERIMENTAL

Materials

Toluene and tetrahydrofuran (THF) were distilled over sodium/benzophenone. *N,N*-dimethyl formamide (DMF) was distilled over CaH₂ under reduced pressure. Other reagents were obtained from commercial resources and used as received. The synthetic details of the cooligomers are depicted in the Supporting Information (SI).

Instruments

¹H-NMR and ¹³C-NMR spectra were recorded on Bruker AV 300-MHz and 400-MHz spectrometer in CDCl₃ with tetramethylsilane (TMS) as an internal standard. Preparative gel permeation chromatography (GPC) purification was carried out with a JAI LC-9104 recycling preparative high performance liquid chromatography (JAIGEL 2H/3H column assembly) with toluene as eluent. Elemental analysis was performed by a Flash EA1112 elemental analysis system. Matrix-assisted laser desorption ionization time-of-flight (MALDI-TOF) mass spectra (MS) was recorded on a Bruker/AutoflexIII Smartbean MALDI Mass Spectrometer with anthracene-1,8,9-triol as the matrix. Solution cyclic voltammetry (CV) measurements were performed on the CHI660a electrochemical analyzer in a solution of 0.1 mol/L tetrabutylammonium hexafluorophosphate (Bu₄NPF₆) in anhydrous CH₂Cl₂. UV-Vis absorption spectra were recorded on a Shimadzu UV3600 spectrometer. Different scanning calorimetry (DSC) measurements were performed on a TA Q100 thermal analyzer at a scanning rate of 10 K/min in N₂. Small angle X-ray scattering (SAXS) was recorded on a Bruker Nanostar instrument operated at 40 kV and 0.65 mA. Transmission electron microscope (TEM) was performed on a JEOL JEM-1011 transmission electron microscope operated at an acceleration voltage of 100 kV. Selected area electron diffraction (SAED) was taken with cameral length of 120 cm and Au (111) diffraction as the external standard.

OSC Device Fabrication and Measurements

ITO (indium tin oxide) glass plates were cleaned with detergent, ultrasonicated in water and dried at 120 °C for 30 min, and then treated with UV/ozone for 15 min. Poly(3,4-ethylene dioxythiophene):poly(styrene sulphonate) (PEDOT:PSS) with a thickness of *ca.* 40 nm was spin-coated on the cleaned ITO at 3000 r/min for 60 s and then baked for 30 min at 120 °C. Then the cooligomer layer with a thickness of *ca.* 70 nm was spin-coated from chlorobenzene solution with a concentration of 12 mg/mL in glovebox. The films were heated at 140 °C for 10 min, or put in CH₂Cl₂ atmosphere for 40 min. Finally, hole blocking-layer *N,N'*-bis(1-ethylpropyl)-3,4,9,10-perylenetetracarboxyl diimide (epPDI, 5 nm), LiF (1 nm) and Al (100 nm) were thermally deposited in a vacuum chamber at a base pressure of less than 4 × 10⁻⁴ Pa. The active area of the device is 12 mm². The devices were sealed in the glovebox before measured in ambient. An Oriel 150W solar simulator with an AM 1.5G filter was used as a light source to produce an intensity of 100 mW/cm² for the illumination of the photovoltaic cells. The light intensity was calibrated by a calibrated silicon diode with KG-5 visible color filter. Current-voltage (*J-V*) characteristics were obtained with a Keithley 236 source meter. External quantum efficiency (EQE) measurements were performed under short-circuit conditions with a lock-in amplifier (SR830, Stanford Research System) at a chopping frequency of 280 Hz during illumination with a monochromatic light from a Xenon lamp.

RESULTS AND DISCUSSION

Synthesis of the Oligomers

F4B-hP, **F5B-hP**, **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP** were synthesized *via* Stille coupling (details please see the SI), and their chemical structures along with the structure of **F5T8-hP** are shown in Fig. 1. The cooligomers were further purified by preparative gel permeation chromatography (PGPC) after the purification by column chromatography on silica gel. Their chemical structures were confirmed by $^1\text{H-NMR}$, $^{13}\text{C-NMR}$, MOLDI-TOF mass spectrometer and elemental analysis. MOLDI-TOF mass spectra of the cooligomers all show a single peak corresponding to the theoretical molecular mass (Figs. S1–S5).

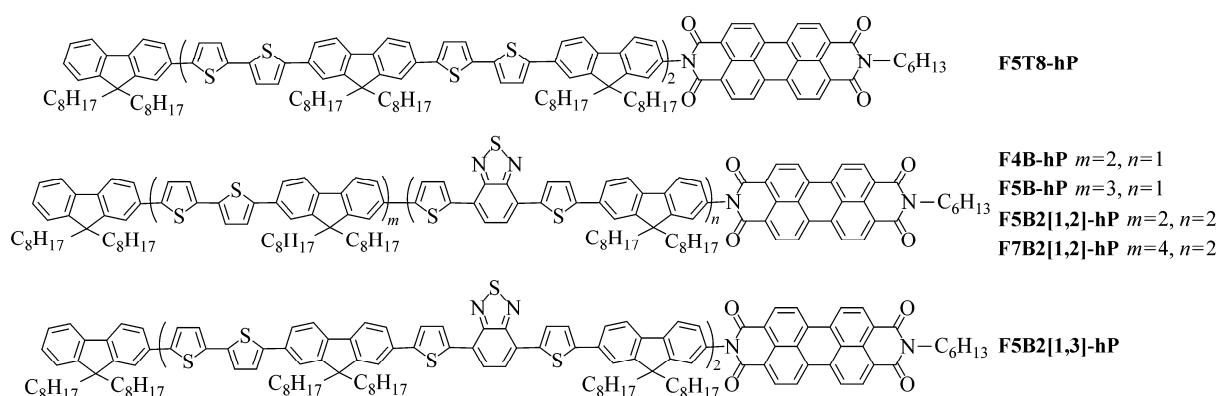


Fig. 1 Chemical structures of the D-A conjugated cooligomers

Photophysical Properties

Solution and pristine film UV-Vis absorption spectra of the cooligomers are presented in Fig. 2. Compared to **F5T8-hP**, the absorption onsets of all the cooligomers show obvious bathochromic shifts in both solution and film states (Table 1), and the absorption in the range of 500–600 nm is greatly enhanced. From solution to film, the absorption onsets of all the cooligomers red-shift *ca.* 50 nm. This may correlate with the more extended chain configuration of the cooligomers in solid state.

Table 1. Absorption maxima, HOMO and LUMO energy levels of the cooligomers

Cooligomer	$\lambda_{\text{max, sol}}$ (nm)	$\lambda_{\text{max, pris}}$ (nm)	HOMO (eV)	LUMO (eV)
F5T8-hP	447, 527	446	−5.26	−3.91
F4B-hP	437, 491, 528	440, 493, 539	−5.27	−3.90
F5B-hP	450, 528	447, 536	−5.26	−3.90
F5B2[1,2]-hP	453, 491, 528	424, 495, 540	−5.32	−3.92
F5B2[1,3]-hP	416, 492, 528	411, 540	−5.28	−3.92
F7B2[1,2]-hP	452, 528	449, 537	−5.24	−3.89

Electrochemical Properties

In order to estimate the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) energy levels of the cooligomers, CV measurements were performed and the corresponding redox curves are displayed in Fig. 3(a). HOMO and LUMO energy levels were calculated by the equations $\text{HOMO} = -(4.80 + E^{\text{ox}})$ eV and $\text{LUMO} = -(4.80 + E^{\text{re}})$ eV, in which E^{ox} and E^{re} represent oxidation and reduction onset potentials, respectively, which were calibrated by the ferrocene/ferrocenium (Fc/Fc $^+$) pair. As shown in Table 1, the cooligomers show almost the same HOMO/LUMO energy levels as **F5T8-hP** (−5.26/−3.91 eV).

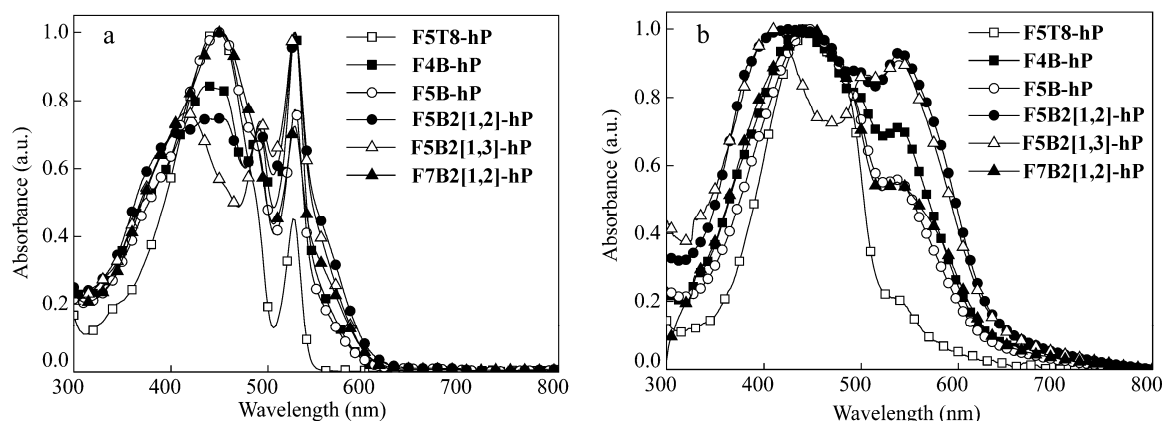


Fig. 2 Solution (a, 5×10^{-6} mol/L in chloroform) and film (b) UV-Vis absorption spectra of **F5T8-hP**, **F4B-hP**, **F5B-hP**, **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP**. Films with a thickness of *ca.* 70 nm were prepared by spin-casting 12 mg/mL chlorobenzene solutions on quartz substrates.

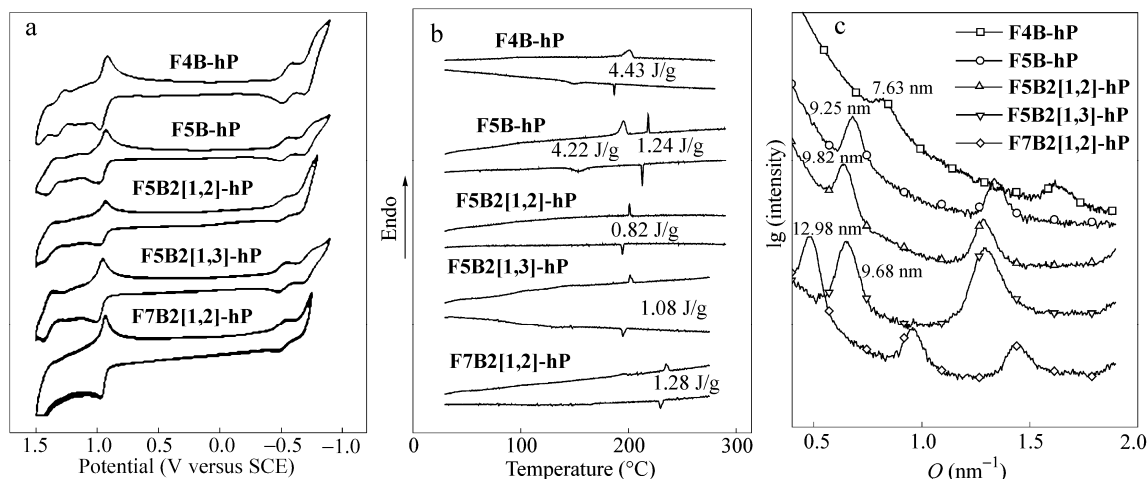


Fig. 3 Cyclic voltammograms in CH_2Cl_2 (10^{-3} mol/L) with Bu_4NPF_6 (0.1 mol/L) as the electrolyte at a scan rate of 50 mV/s (a), the first cooling and the second heating DSC traces at a heating/cooling rate of 10 K/min in N_2 (b), and powder SAXS after thermally annealed at 140 °C for 10 min (c) of the cooligomers

Thermal Properties

The thermal properties of the cooligomers were evaluated by DSC with a heating/cooling rate of 10 K/min in N_2 atmosphere. The first cooling and the second heating DSC scans are given in Fig. 3(b). In the heating scan, **F4B-hP** shows a mesophase-isotropic state transition (T_i) at 201.0 °C. **F5B-hP** has an additional transition attributed to the phase transition between different liquid crystalline phases. The introduction of BT unit lowers the T_i of the cooligomers. For instance, **F5B-hP** shows a T_i of 218.2 °C, 30.3 °C lower than that of **F5T8-hP** (248.5 °C). For **F5B2[1,2]-hP** and **F5B2[1,3]-hP** which contain two BT units, T_i values further decrease to 200.9 and 201.0 °C, respectively. **F7B2[1,2]-hP** has the highest T_i (234.7 °C) due to its longer conjugated length.

Morphological Characterization

To evaluate the packing structures of the cooligomers in solid state, the thermally annealed powders of the cooligomers were investigated by SAXS. As shown in Fig. 3(c), all the cooligomers can form lamellar packing structure with the (001) d -spacings of 7.63, 9.25, 9.82, 9.68 and 12.98 nm for **F4B-hP**, **F5B-hP**, **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP**, respectively. These values are close to the molecular lengths of the

corresponding cooligomers calculated from MM2 method (7.80, 9.16, 10.25, 9.46 and 12.00 nm, respectively).

Thin films of the cooligomers with the thickness of *ca.* 70 nm on PEDOT:PSS covered glass slides were floated with water to micro-grid for TEM characterization. The pristine films of the cooligomers were all featureless (Fig. S6). After thermal annealing at 140 °C for 10 min, dark-bright stripes appeared in the films of **F4B-hP**, **F5B-hP** and **F7B2[1,2]-hP** (Fig. 4). However, the order of the films was still relatively low. After CH₂Cl₂ vapor annealing for 40 min, more ordered stripes emerged in the films of **F4B-hP**, **F5B-hP** and **F7B2[1,2]-hP**, and lamellar nanostructures also appeared in the films of **F5B2[1,2]-hP** and **F5B2[1,3]-hP**. The periods of the lamellae are 7.2, 9.1, 9.6, 9.5 and 12.5 nm for **F4B-hP**, **F5B-hP**, **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP**, respectively, which are in accordance with the SAXS measurements as shown in Fig. 3(c). Compared with the films of **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP**, which contain two BT units, the films of **F4B-hP** and **F5B-hP** show higher order, which is further confirmed by the observation of clear diffraction rings in SAED patterns. Moreover, for cooligomers containing two BT units, the film order is in a sequence of **F7B2[1,2]-hP** > **F5B2[1,3]-hP** > **F5B2[1,2]-hP**, as indicated by TEM images (Figs. 4h, 4i and 4j). To understand why **F4B-hP** and **F5B-hP** can afford the films with higher order than **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP**, we carried out computer simulation of the backbone configuration of the D segments in **F5B-hP**, **F5B2[1,2]-hP** and **F5B2[1,3]-hP**, which are named as **F4B**, **F4B2[1,2]** and **F4B2[1,3]**. To simplify the calculation, octyl substituents were replaced by CH₃ groups. As shown in Fig. 5, the introduction of two BT units causes the severe distortion of the backbone of the segments with the bending angle of 130° and 148° for **F4B2[1,2]** and **F4B2[1,3]**, respectively. This severe distortion may prohibit **F5B2[1,2]-hP** and **F5B2[1,3]-hP** molecules from ordered packing, resulting in low ordered films. **F4B2[1,3]** exhibits less distorted backbone than **F4B2[1,2]**, which is consistent with that **F5B2[1,3]-hP** film exhibits higher order than that of **F5B2[1,2]-hP**, as shown in Fig. 4.

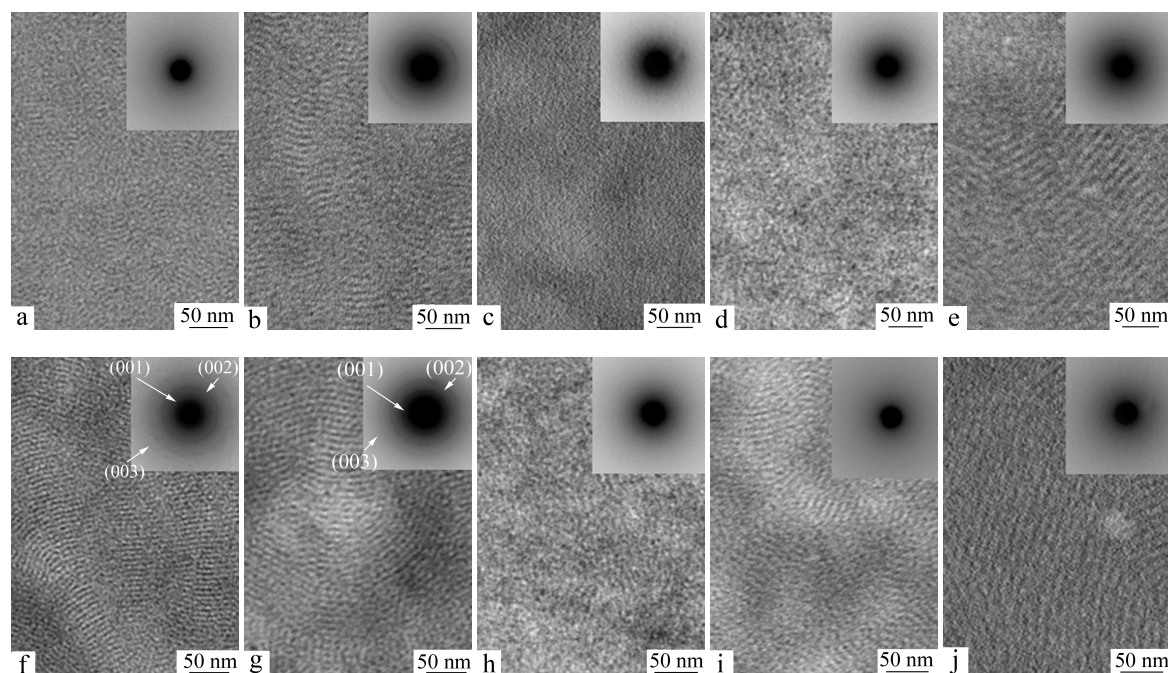


Fig. 4 TEM images of **F4B-hP** (a, f), **F5B-hP** (b, g), **F5B2[1,2]-hP** (c, h), **F5B2[1,3]-hP** (d, i) and **F7B2[1,2]-hP** (e, j) films; thermal annealed (a–e) at 140 °C for 10 min and CH₂Cl₂ vapor annealed for 40 min (f–j). Insets on top right corner of the images are corresponding SAED patterns of the films.

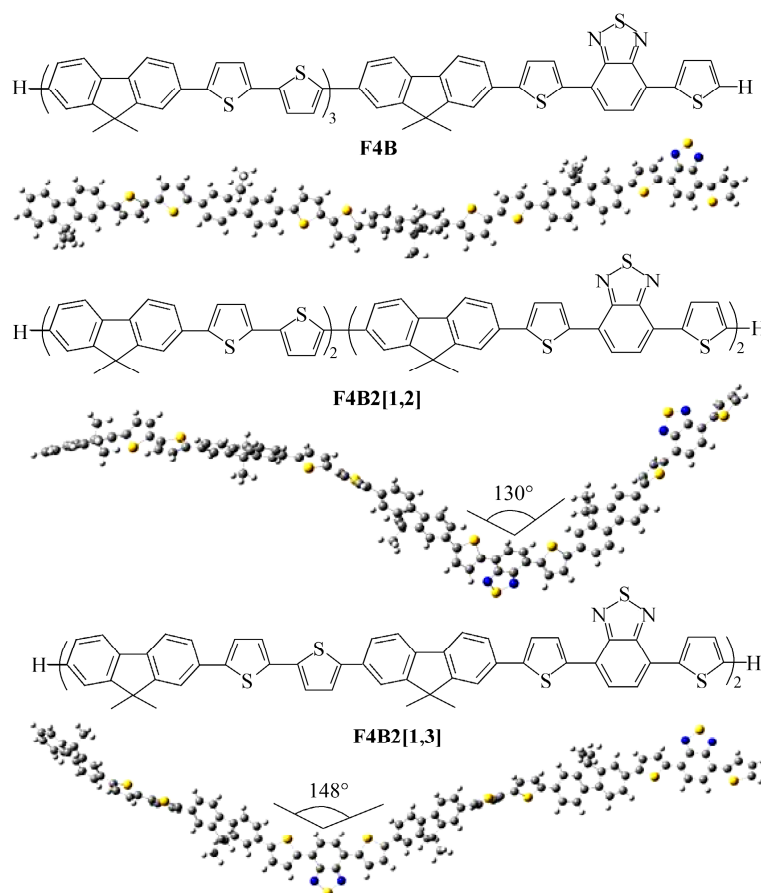


Fig. 5 Chemical structures and optimized geometry of **F4B**, **F4B2[1,2]** and **F4B2[1,3]** from DFT calculation using **B3LYP/6-31G (d)** by Gaussian 09. The dihedral angles between thiophene and fluorene units and thiophene and BT units are 49° and 31°, respectively.

Photovoltaic Properties

Photovoltaic properties of the cooligomers were investigated with a device structure of ITO/PEDOT:PSS/cooligomer/epPDI/LiF/Al, and the thickness of the cooligomer layers was *ca.* 70 nm. A thin layer of epPDI (*ca.* 5 nm) was incorporated as a hole blocking layer, resulting in noticeable improved FF and J_{SC} ^[22]. The performance parameters of the photovoltaic devices are listed in Table 2. Consistent with the poor morphology of the films (Fig. S6), the devices based on the pristine films all showed poor device performance. The J - V curves and EQE profiles of the devices based on the thermally annealed and CH₂Cl₂ vapor annealed films are displayed in Fig. 6. **F4B-hP**, **F5B-hP**, **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP** all show photo-response in the range of 350–630 nm, which is consistent with their film absorption spectra (Fig. S7) and is 50 nm broader than **F5T8-hP**^[22]. **F4B-hP**, which has the shortest molecular length, shows PCE of 1.15% and 1.28% when the films were thermally and solvent vapor annealed, respectively. As the molecular length increases, the solvent vapor annealed film of **F5B-hP** gives higher device performance, with a PCE up to 1.72%. The J_{SC} and FF increased from 5.14 mA/cm² and 0.29 for **F4B-hP** to 5.76 mA/cm² and 0.34 for **F5B-hP**, which is in accordance with the previous investigation^[22, 23]. In the previous report^[22], **F5T8-hP** exhibited a PCE of 1.50%. The enhancement of PCE of **F5B-hP** is attributed to higher J_{SC} for its more efficient harvesting of light. Even upon thermal annealing, **F5B-hP** can still affords a PCE of 1.56% (V_{OC} = 0.87 V, J_{SC} = 5.49 mA/cm², FF = 0.33). Although **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP** show stronger absorption at 500–600 nm than **F4B-hP** and **F5B-hP**, they exhibit lower PCEs ascribed to lower current densities. Based on aforementioned

discussion on film morphology, their poorer device performance can be attributed to the lower order of the films owing to their distorted backbone. Of these three cooligomers, PCE increased from **F5B2[1,2]-hP** to **F5B2[1,3]-hP** and then to **F7B2[1,2]-hP**, which is consistent to the film morphology observation by TEM (as shown in Fig. 4).

Table 2. Solar cell performance of the cooligomers^a

Cooligomer		V_{OC} (V)	J_{SC} (mA/cm ²)	FF	PCE (%)
F4B-hP	pris	0.83	0.71	0.23	0.13
	TA	0.90	4.46	0.29	1.15
	SA	0.86	5.14	0.29	1.28
F5B-hP	pris	0.84	0.90	0.26	0.20
	TA	0.87	5.49	0.33	1.56
	SA	0.87	5.76	0.34	1.72
F5B2[1,2]-hP	pris	0.15	7.5×10^{-3}	0.30	3.3×10^{-3}
	TA	0.84	1.93	0.24	0.40
	SA	0.87	2.69	0.26	0.60
F5B2[1,3]-hP	pris	0.72	0.57	0.25	0.10
	TA	0.88	2.51	0.27	0.59
	SA	0.87	3.86	0.29	0.97
F7B2[1,2]-hP	pris	0.72	0.48	0.27	0.094
	TA	0.84	4.26	0.33	1.18
	SA	0.84	4.56	0.30	1.14

^a pris = pristine; TA = thermal annealed; SA = solvent vapor annealed

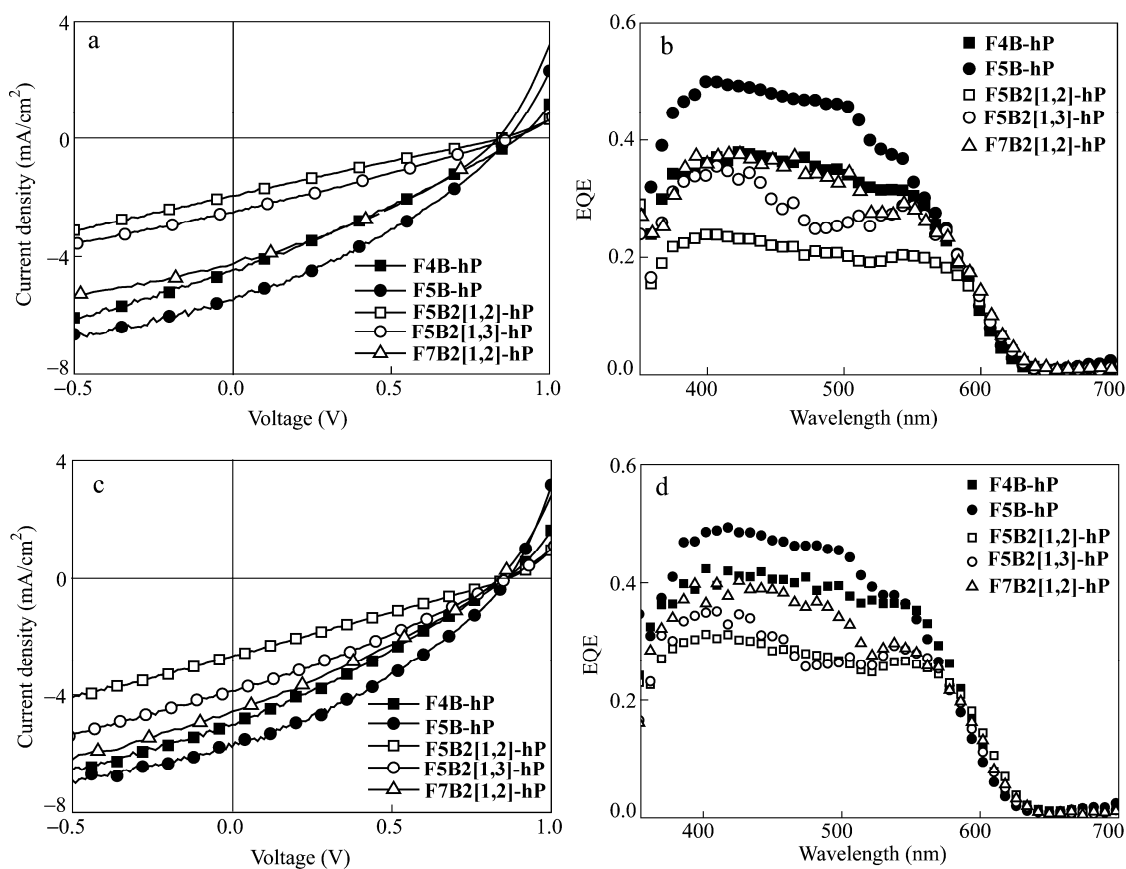


Fig. 6 J - V characteristics (a, c) and EQE curves (b, d) of the solar cells based on thermal annealed (a, b) and CH_2Cl_2 vapor annealed (c, d) films of the cooligomers

CONCLUSIONS

We have synthesized five novel donor-acceptor (D-A) conjugated cooligomers, *i.e.*, **F4B-hP**, **F5B-hP**, **F5B2[1,2]-hP**, **F5B2[1,3]-hP** and **F7B2[1,2]-hP**, which show absorption spectra covering the range of 300–630 nm. Upon solvent vapor annealing, all the cooligomers can form films characterized with lamellar nanostructures with the periods relative to the molecular lengths. The order of the nanostructured films greatly depends on the backbone configuration. The cooligomers containing two 2,1,3-benzothiadiazole (BT) units have more distorted backbone, therefore can only form the nanostructured films with relatively lower order. Accordingly, the cooligomers exhibited distinct photovoltaic device performance. Of the cooligomers, **F5B-hP** exhibited the best device performance, PCEs of 1.56% and 1.72% were demonstrated for the devices based on thermally and solvent vapor annealed films of **F5B-hP**, respectively. Our results imply that efficient single molecule solar cells can be fabricated with D-A cooligomers while their backbone is carefully designed.

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